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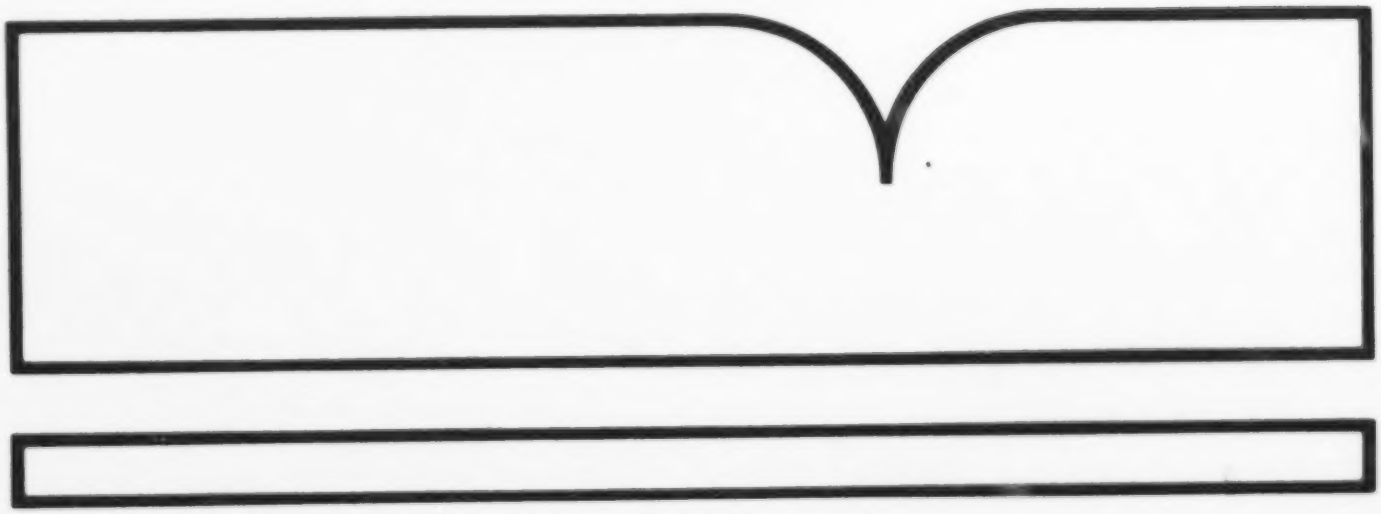

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16. ABSTRACT

Coagulation of Mississippi River water improved the removal of total organic carbon (TOC) and trihalomethane precursors by granular activated carbon. This increased removal is thought to be due to the reductions in TOC and in poorly adsorbed higher-molecular-weight organics that are achieved by coagulation. A cost analysis indicated that very high coagulant dosages would be needed during pretreatment to minimize the overall costs of chemical treatment.

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Influence of Coagulation on Removal of Organics by Granular Activated Carbon

Michael J. Semmens, Alan B. Staples, Gary Hohenstein, and Gregor E. Norgaard

Coagulation of Mississippi River water improved the removal of total organic carbon (TOC) and trihalomethane precursors by granular activated carbon. This increased removal is thought to be due to the reductions in TOC and in poorly adsorbed higher-molecular-weight organics that are achieved by coagulation. A cost analysis indicated that very high coagulant dosages would be needed during pretreatment to minimize the overall costs of chemical treatment.

Although coagulation is traditionally a pretreatment process for removing particulates, many investigators have demonstrated that it can also reduce the concentration of naturally occurring organics.[1] Its ability to reduce organics, however, is limited, and if unacceptably high concentrations remain, activated carbon must be employed.

Previous investigators have identified the benefits of coagulation as a pretreatment for activated carbon adsorption.[2-4] Randtke and Jepsen[2] concluded that the formation of aluminum-organic complexes was the major contributing factor to improved organics adsorption.

The exact mechanism by which alum improved the adsorption capacity of carbon was not clear. Part of the difficulty in explaining the observed behavior was the fact that aluminum did not improve carbon adsorption capacity until a certain alum dose had been applied. The effective dose was somewhat less than the stoichiometric requirement for systems showing type 1 behavior or sufficient to cause a significant amount of total organic carbon (TOC) reduction (30 percent) in systems exhibiting type 2 behavior.[2] The characteristic dose responses of type 1 and type 2 waters are represented in Figure 1.

Lee et al[3] tested the influence of alum pretreatment on the adsorption behavior of peat fulvic acid and concluded that alum coagulation enhanced both carbon adsorption capacity and the rate of uptake. However, increased capacity was thought to be the major contribution to longer carbon column service life.

An earlier study[4] indicated that coagulation of Rhine River water (following bank filtration and ozonation) improved adsorption. Residual organics were better adsorbed than the original organics in the water. In addition, these authors observed that the adsorbability of the residual organics was better following aluminum sulfate coagulation than following ferric chloride pretreatment.

Experimental procedures

Water source. The water used in this study (Table 1) was extracted from the Mississippi River at Minneapolis, Minn., close to the water treatment plant intake.

River water samples were collected in 20-L glass carboys and then rough filtered by gravity flow through 2.9 ft (0.9 m) of filter sand (0.4-mm effective

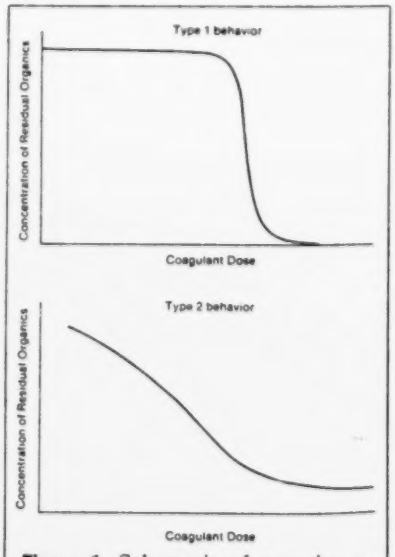

Figure 1. Schematic of organics removal as a function of coagulant dose in waters exhibiting type 1 and type 2 behavior[2]

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ze). The sand was supported in a 3.9-4.- (10-cm-) diameter glass column by 7 in. (12 cm) of graded gravel on a pecially constructed glass diffuser plate. ll tubing and connections were PTFE. he filter effluent was pumped directly nto a 200-L, refrigerated, stainless steel ank. When about 50 gal (189 L) of iltered water had been pumped into the ank, the refrigeration system was urned on. The contents of the tank were nixed with a stainless steel impeller and were frozen from the outside toward the inside, a technique developed by Shapiro.[5,6] In about 32 h the concentrate, with 90-95 percent of the original organic content of the river water,[7] was reduced to about 7 gal (28 L). This concentrate was removed and stored at 4°C in a stainless steel drum. Samples of the concentrate were later used to reconstitute river water of constant quality by adding organics-free distilled water in the correct proportion.

Calcium carbonate and sulfates precipitated from the concentrate after long storage periods. Although no significant change occurred in the organic content or character (as measured by removal behavior) of the concentrates, unconcentrated samples were tested. Tests were conducted as rapidly as possible to minimize storage time.

Coagulation tests. Reconstituted and fresh river water samples were treated according to a modified jar test technique.[7] A gang stirrer* was employed for flocculation, while a separate high speed stirrer equipped with a PTFE propeller was used for the rapid mix. The desired volumes of stock coagulant solutions were injected into the vortex created by the rapid mixer and were mixed for 5-10 s. The speed of rotation was then reduced to 30 rpm, and acid or base (1*N* sulfuric acid [H_2SO_4] or 1*N* sodium hydroxide [NaOH]) was added for pH adjustment.

Following 30 min of flocculation at 30 rpm, the samples were settled for approximately 15 min, filtered through prewashed filter paper, and stored for later analysis at 4°C. Earlier studies had indicated that this type of filter paper simulated typical rapid sand filtration at 2 gpm/sq ft (4.9 m/h). Effluent water samples from a 2.9 ft- (0.9-m-) deep rapid sand filter and from the filter paper contained identical concentrations of organics as measured by ultraviolet absorbance (UVA) and TOC. When turbidity measurements were required, 1 h of sedimentation was provided prior to filtration. Sedimentation time did not influence organics removal.

Alum—analytical grade aluminum sulfate [$Al_2(SO_4)_3 \cdot 18\ H_2O$]—was used as the coagulant. A measured volume of a 50-g/L stock alum solution was injected during rapid mix to provide coagulation.

Carbon adsorption studies. Samples (26.4 gal [100 L]) of river water were coagulated and flocculated in a stainless steel tank, settled, and filtered through a trimedia filter before use in the adsorption study. The filtered water was then pumped to small-scale (0.3 × 11.8 in. [0.9 × 30 cm]) glass columns packed with 2.3-3.5 in. (6-9 cm) of carbon† at a flow rate of between 3 and 4 mL/min. Empty bed contact times were approximately 1.1-1.3 min. The experimental design has been described in detail previously.[8]

Analyses. Total organic carbon was measured with an analyzer.‡

Ultraviolet absorbance was monitored using an ultraviolet-visible spectrophotometer,§ which measured absorbance in a 5-cm quartz cell at 254 nm. Samples were brought to pH 4.0 with H_2SO_4 prior to UVA measurement.

TABLE 1
Composition of Mississippi River water sample

Parameter	Measurement
TOC	5-10 mg/L
Alkalinity	90-140 mg $CaCO_3$/L
Ca^{2+}	35-45 mg/L
Mg^{2+}	8-12 mg/L
UVA	0.50-1.20
pH	8.3

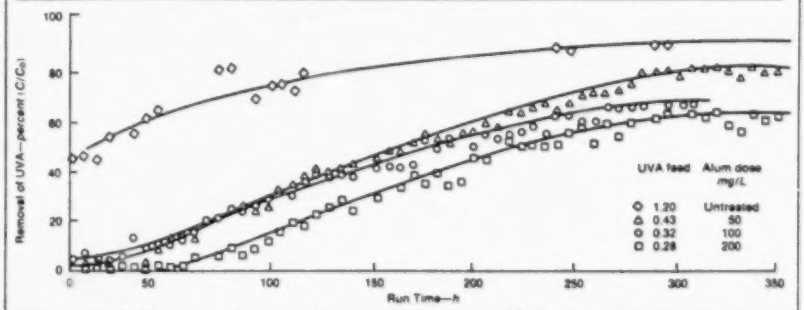

Figure 2. Influence of alum dose on the percentage removal of UVA from Mississippi River water by activated carbon *(pH of coagulation—5.0; pH of adsorption—7.0; EBCT—1.15 min; flow rate—3.5 mL/min)*

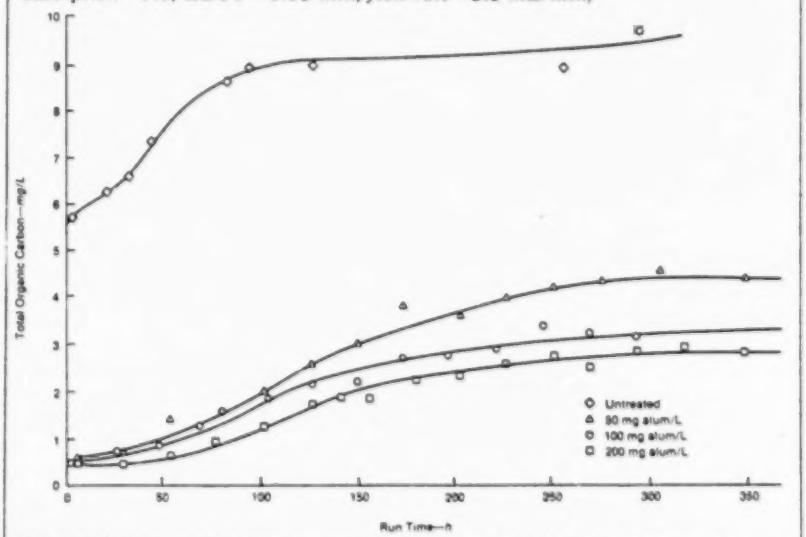

Figure 3. Influence of alum dose on the removal of TOC from Mississippi River water by activated carbon *(pH of coagulation—5.0; pH of adsorption—7.0; EBCT—1.15 min; flow rate—3.5 mL/min)*

Trihalomethane formation potential was used to quantify precursor concentration. Water samples were chlorinated for 48 h with excess free chlorine, and the resulting THMs were measured by solvent extraction and gas chromatography** using an electron capture detector, a technique previously described by Semmens and Field.[7]

Results

To evaluate the effectiveness of coagulation, three granular activated carbon (GAC) columns were operated with pre-

*Phipps and Bird, Richmond, Va.
†50 × 80 Nuchar WV-G, Westvaco, Corington, Va.
‡Barnstead Co., Boston, Mass.
§Beckman Instruments, Fullerton, Calif.
**HP 5840A, Hewlett Packard, Palo Alto, Calif.

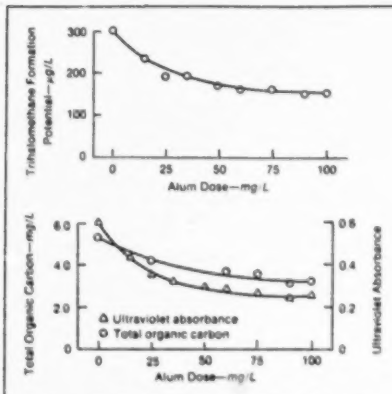

Figure 4. Influence of alum dose on organics removal from river water sample C *(pH of coagulation—5.0)*

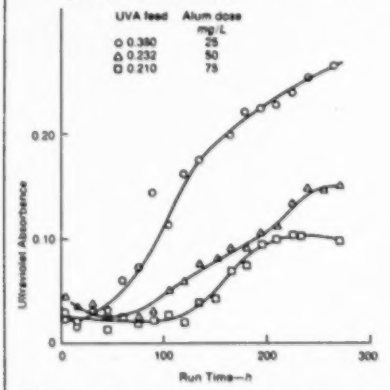

Figure 5. Influence of alum dose on removal of UVA from river water sample C by activated carbon *(pH of coagulation—5.0; pH of adsorption—5.0; EBCT—1.15 min; flow rate—3.5 mL/min)*

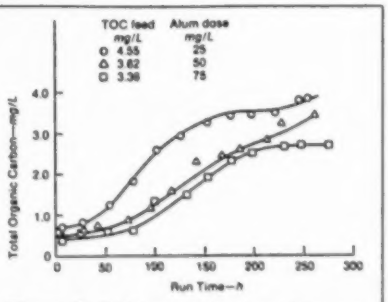

Figure 6. Influence of alum dose on removal of TOC from river water sample C by activated carbon *(pH of coagulation—5.0; pH of adsorption—5.0; EBCT—1.15 min; flow rate—3.5 mL/min)*

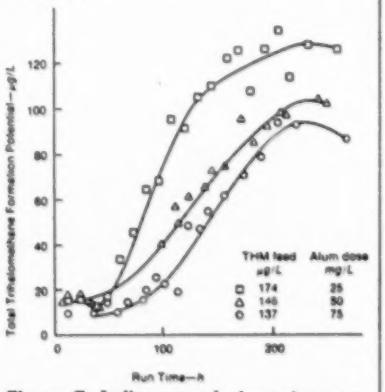

Figure 7. Influence of alum dose on removal of THM precursors from river water sample C by activated carbon *(pH of coagulation—5.0; pH of adsorption—5.0; EBCT—1.15 min; flow rate—3.5 mL/min)*

TABLE 2

Molecular weight distribution of organics in Mississippi River water before and after coagulation[a]

Nominal Molecular Weight Range	TOC—*percent*	
	Untreated Water	Water Coagulated With Alum
<1K	23	56
1-10K	50	38
10-100K	10	0
>100 K	17	6

treated water and a fourth (the control) with untreated river water. A sample of reconstituted river water with 10.7 mg TOC/L was coagulated with alum doses of 50, 100, and 200 mg/L at pH 5.0. Following sedimentation and filtration, the pH was adjusted to 7.0. The results obtained when the water samples were applied to the GAC columns are plotted in Figures 2 and 3.

Figure 2 shows the breakthrough data as percentage removals of organics based on UVA measurements versus time. The data clearly demonstrate the improved performance following coagulation of the river water. Without pretreatment the organics broke through the activated carbon rapidly, and low effluent UVA values could not be achieved. As the coagulant dose was increased, improved removal by the activated carbon was observed. Although the organics in the influent decreased as the level of pretreatment increased, the percentage removal of organics across the GAC columns improved. The breakthrough curves in Figure 2 are quite similar. They were displaced to the right as the level of pretreatment was increased.

Figure 3 shows the same trend in the data for TOC removal. Greater than 50 percent breakthrough of TOC occurred immediately after the GAC columns were put in service for untreated river water. The effect of coagulation was dramatic, with removal of TOC improving as the coagulant dose was increased.

Although the results showed the merit of improving organics removal by coagulation pretreatment, the differences in performance for samples receiving different coagulant dosages were not as exaggerated as had been anticipated. Coagulant dosages were based on the levels of organics removal expected as a result of earlier jar tests, but, as shown in Figures 2 and 3, the pretreated waters were not very different in organic content, and the levels of pretreatment were quite similar.

Additional coagulation-adsorption studies were conducted on freshly collected river water samples. Two data sets were collected, yielding similar results. The river water sample contained about half the TOC (5.4 mg/L) present in the reconstituted water tested in the first study. A jar test study revealed that, at pH 5.0, up to 60-percent UVA removal could be obtained during coagulation and that the removals were dose dependent (Figure 4). Removals of turbidity were excellent for coagulant doses >30 mg/L. On the basis of these tests, coagulant doses of 25, 50, and 75 mg/L were selected for pretreatment. Results of the study of adsorption at pH 5.0 (run C) are presented in Figures 5-7, which show the trends observed in the earlier study, with carbon column performance improving with greater levels of pretreatment. Trihalomethane precursor removal also improved as the coagulant dose was increased.

Discussion

The large initial breakthrough of TOC observed for untreated river water (Figure 3) was not simply a kinetic effect resulting from the column size used. Studies have shown that breakthrough curves obtained with minicolumns compare favorably with curves obtained with a 3.2-ft- (1-m-) deep carbon bed (12 × 20 mesh) loaded at 3.6 gpm/sq ft (9 m/h). Although the dimensionless breakthrough curves are not superimposed on each other perfectly, the same initial TOC values are obtained, and the curves are close enough in character to make the minicolumn data valuable in assessing different operating conditions.

Coagulation can remove a large fraction of the naturally occurring organics in Mississippi River water. Much of the improvement in GAC performance can be attributed to the lowered organic content of the water resulting from the increased coagulant dosage. This effect is apparent when the service time for a specified treatment objective is plotted against the concentration of influent organics for coagulated waters. For example, the curve shown in Figure 8 is obtained when a treatment objective of 1

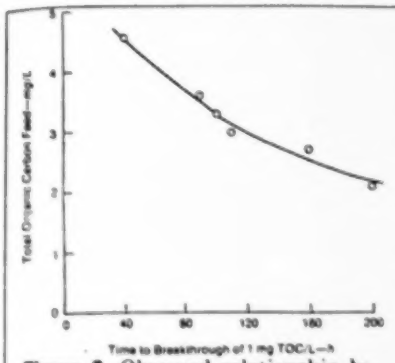

Figure 8. Observed relationship between influent TOC and carbon service life for a specified treatment objective of 1 mg TOC/L *(pH of adsorption—5.0)*

TABLE 3

Relationship between coagulant dose and service time for breakthrough of 1 mg TOC/L at pH 5.0

Coagulant Dose *mg/L*	Breakthrough Time *h*
25	40
50	80
75	100
100	112

TABLE 4

Data employed in the calculation of optimum coagulant dose

Parameter	Value
B (cost of coagulant)	\$0.30/kg*
Q (flow rate)	5.04 L/d
V (volume of carbon bed)	5.73×10^{-6} m^3
M (cost of carbon)	\$1000/m^3†

*Cost of bulk shipment to Minneapolis (data from municipal water treatment plant, November 1981)
†Cost of bulk shipment to Minneapolis (data courtesy of Westvaco, Corington, Va., November 1981)

mg TOC/L is selected. The major difference between the breakthrough curves for untreated and coagulated waters cannot be explained, however, by the differences in influent TOC alone. The initial concentration of TOC in the GAC effluent for all the coagulated samples was approximately 0.5 mg/L. However, for untreated water it was an order of magnitude higher. This suggests that the coagulation process modifies the character of the organic content in such a way that the residual organics exhibit improved adsorption behavior.

Earlier studies on Mississippi River water[7,9,10] demonstrated conclusively that alum coagulation effectively removes higher-molecular-weight organic compounds. Typical molecular weight distributions before and after coagulation are presented in Table 2. The residual organics were classified[9] as predominantly hydrophilic and acidic and neutral in character using the Leenheer-Huffman fractionation procedures.[11] The ability of coagulation to remove the higher-molecular-weight compounds probably contributes to the improved adsorption of the residual organics, because many investigators have observed that higher-molecular-weight compounds are not well removed by activated carbon.[3,12] Lee et al[3] also observed that the higher-molecular-weight fraction of a peat fulvic acid exhibited the same adsorption behavior as the unfractionated material. This suggested that the higher-molecular-weight fraction controlled the observed adsorption behavior in the unfractionated fulvic acid. The authors invoked an association between the lower- and higher-molecular-weight fractions in the unfractionated mixture to explain this behavior. Thus, coagulation removes the weakly adsorbing high-molecular-weight fraction of the organic content and leaves the strongly adsorbing low-molecular-weight fraction in solution. The lower average molecular weight of the organics in solution following coagulation will also improve adsorption kinetics.[3]

In the current study, the major improvement in GAC performance resulted from the combined benefits of TOC reduction and the removal of weakly adsorbing high-molecular-weight organics. The possibility of an association between the added aluminum and the residual organic content of the water, as suggested by Randtke,[2] was not investigated. However, parallel pretreatment studies of lime softening indicated that when the level of organics removal during pretreatment was the same, the adsorption performances with softened and coagulated waters were comparable.[13] Softening, like alum coagulation, removed higher-molecular-weight organic compounds from the river water sample, causing a general improvement in GAC performance. This suggests that if there was an interaction between aluminum and the residual organics, the product was no better adsorbed than the organics remaining in solution after chemical softening.

Optimum coagulant dose

The impact of coagulation was evaluated over a one-year period with three different river water samples. In each case the dose response of the water for organics removal by coagulation was different. For one sample, removals increased up to an alum dose of >200 mg/L; another sample showed a plateau in organics removal at about 90 mg/L; and the third sample exhibited intermediate behavior. Nevertheless, the adsorption behavior of the organic content of each water sample was improved to a similar extent by coagulation pretreatment, and the service time to 1 mg TOC/L breakthrough could be correlated with the TOC in the carbon column feed, as shown in Figure 8 for an adsorption pH of 5.0. The useful life of a carbon column can, therefore, be correlated indirectly with the coagulant dosage employed during pretreatment for a specific water sample at a fixed pH. For example, it is possible to correlate service time and coagulant dosage for the water sample tested in run C by using the jar test data in Figure 4 and the correlation in Figure 8. The data presented in Table 3 were extracted in this way. These data were modeled with an equation of the form $\ln (D/Do) = kT$, in which k and Do are constants with values equal to 0.0193 and 11.3, respectively; D is the coagulant dose (milligrams per litre); and T is the breakthrough time (hours).

This type of correlation can be used to identify the optimum degree of pretreatment that will yield the desired water quality at a minimum cost. The optimum dosage can be calculated if the total operating cost of treatment is assumed to be equal to the chemical costs for coagulation plus the cost of carbon replacement for the analysis. Power costs and capital recovery costs are assumed to be independent of the coagulant dosage for the dosages considered.

The coagulation cost (\$/d) can thus be represented by an expression of the type:

$$C_{COAG} = DQB \times 10^{-6} \qquad (1)$$

in which D = coagulant dose (mg/L), Q = flow rate (L/d), and B = cost of coagulant (\$/kg).

The carbon cost (\$/d) can be similarly estimated:

$$C_{GAC} = (24\ VM)/T \qquad (2)$$

in which V = volume of carbon bed (m^3), M = cost of carbon (\$/m^3), and T = service time to breakthrough of 1 mg TOC/L (h).

$$\text{Total cost} = C_{COAG} + C_{GAC} = C_T$$

$$C_T = DQB \times 10^{-6} + (24\ VM)/T \qquad (3)$$

For adsorption at pH 5.0, the service time can be expressed in terms of coagulant dose, and therefore

$$C_T = DQB \times 10^{-6} + [24\,(0.0193)\,VM]/[\ln\,(D/Do)] \quad (4)$$

By differentiating and simplifying Eq 4, the following expression is obtained:

$$dC_T/dD = QB \times 10^{-6} - 0.463\,(VM/\,D[\ln\,(D/Do)]^2) \quad (5)$$

Thus the coagulant dose required to minimize treatment costs is given by setting Eq 5 to zero and rearranging it:

$$D[\ln\,(D/Do)]^2 = 4.63 \times 10^5\,(VM/QB) \quad (6)$$

The optimum coagulant dose at pH 5.0 can by calculated using Eq 6 and will depend on the relative costs of the carbon and the coagulant. As the ratio of carbon cost to coagulant cost increases, the optimum coagulant dose will also increase. The optimum dose is also apparently related to V/Q, or the empty bed contact time (EBCT). However, the relationship between service life and coagulant dose was developed from operational data, and it is important to recognize that the EBCT is not a variable in this equation. The dose-service time relationship shown in Figure 8 is strongly dependent on operating conditions. Solution pH and contact time with the carbon will have a pronounced effect.

By substituting actual operating data from the small-scale-column study and cost data (November 1981) as listed in Table 4, the optimum coagulant dose was calculated to be 208 mg alum/L. Because this coagulant dose was outside the range studied, the result is suspect. Nevertheless, it indicates that a very high level of pretreatment is required to minimize the overall cost of treatment. This result is dependent on an objective of 1 mg TOC/L. A similar analysis for a treatment objective of <2 mg TOC/L indicated that the optimum dose would only drop to 145 mg alum/L.

The above analysis does not include a cost function for alum sludge disposal, which represents a major cost of pretreatment.[11] To assess the impact of sludge disposal costs on the optimum coagulant dose, it was assumed that sludge disposal would double the effective cost of the coagulant. The recomputed optimum doses for objectives of 1 mg TOC/L and 2 mg TOC/L were 140 and 97 mg/L, respectively.

This analysis is also deficient in assessing the impact of solution pH on pretreatment and carbon performance. Solution pH is extremely important for several reasons:

- Coagulants such as alum and ferric chloride are acidic and tend to reduce the solution pH. The extent of pH reduction depends on the coagulant dosage rate and the alkalinity of the water.
- The final solution pH attained during coagulation strongly influences the extent of organics removal during coagulation. For example, studies[7] have demonstrated that TOC and THM precursors were reduced by about 60 percent at pH 5.0 but were reduced by only 15 percent at pH 8.0 when Mississippi River water was coagulated with 25 mg alum/L. Superior organics removal at the lower operating pH results in less TOC being pumped to the GAC columns and extends the useful life of the carbon.
- Solution pH has a major impact on the adsorption capacity of GAC. Semmens et al[8] clearly demonstrated that if river water samples coagulated under identical conditions (and therefore having the same organic content) were adjusted to different pH values prior to GAC treatment, the GAC removed more organics for a longer period of time under acidic conditions. For example, at pH 8.0 a GAC column treated approximately 3100 bed volumes before 1 mg TOC/L broke through in the effluent, but if the solution pH was reduced to 5.0, more than 12 000 bed volumes were treated prior to breakthrough (influent TOC = 2.5 mg/L).

The analysis of the impact of coagulant dosage presented in this article was conducted at a fixed pH of 5.0 during both coagulation and adsorption. If the same study had been conducted at a different operating pH, a different optimum coagulant dose would have been calculated. Also, the impact of coagulant dosage on GAC performance would have been greater than shown in this analysis if coagulation had been conducted without pH control because of the combined reductions in organic content and pH.

Nevertheless, the concept of an optimum coagulant dose has been demonstrated. The coagulant dosages required to optimize the cost of water treatment by GAC are typically higher than those for normal turbidity removal.

Conclusions

Coagulation pretreatment of Mississippi River water improved the performance of GAC for TOC and THM precursor removal (on the basis of effluent organics breakthrough as well as percentage removal).

The improved removal of organics after pretreatment is thought to be due to the combined effects of reduced influent TOC and the removal of poorly adsorbed higher-molecular-weight organics.

A simple cost analysis indicated that very high coagulant dosages were required during pretreatment to minimize the overall costs of chemical treatment.

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